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IS 1448-70 (1968): Methods of test for petroleum and its products, Part 70: Residue in liquefied petroleum gases [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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(Reaffirmed 2003)
Adopted: *22 November 1968*

RESIDUE IN LIQUEFIED PETROLEUM GASES

[P : 70]

(Second Reprint MARCH 1984)

Adapted from ASTM Designation D 2158-65

1. SCOPE

1.1 This method describes a procedure for determining extraneous materials weathering above 38°C which are present in liquefied petroleum gases (LPG).

2. OUTLINE OF THE METHOD

2.1 A 100-ml sample of LPG is weathered in a 100-ml centrifuge tube. The volume of residue remaining at 38°C is measured and recorded, as also the appearance of a filter paper to which the residue has been added in measured increments.

3. TERMINOLOGY

3.0 For the purpose of this method, the following definitions shall apply.

3.1 Residue — The volume, measured to the nearest 0·05 ml, of hydrocarbon boiling above 38°C resulting from evaporation, under specified conditions, of 100 ml of sample.

3.2 R Number (Residue Number) — Volume of residue multiplied by 200.

3.3 Oil Stain Observation — The volume of solvent-residue solution required to yield an oil ring that persists for 2 minutes under specified conditions on a prescribed filter paper.

3.4 O Number (Oil Number) — Number obtained on dividing 10 by the oil stain observation.

4. APPARATUS AND MATERIALS

4.1 Centrifuge Tube — The tube (*see* Fig. 1) shall be made of glass and shall be of 100 ml capacity. It shall be cone shaped, conforming to the dimensions shown in Fig. 1 and shall be of 1·5 to 2 mm wall thickness to weigh not less than 60 g. Its graduations shall be distinct and shall read

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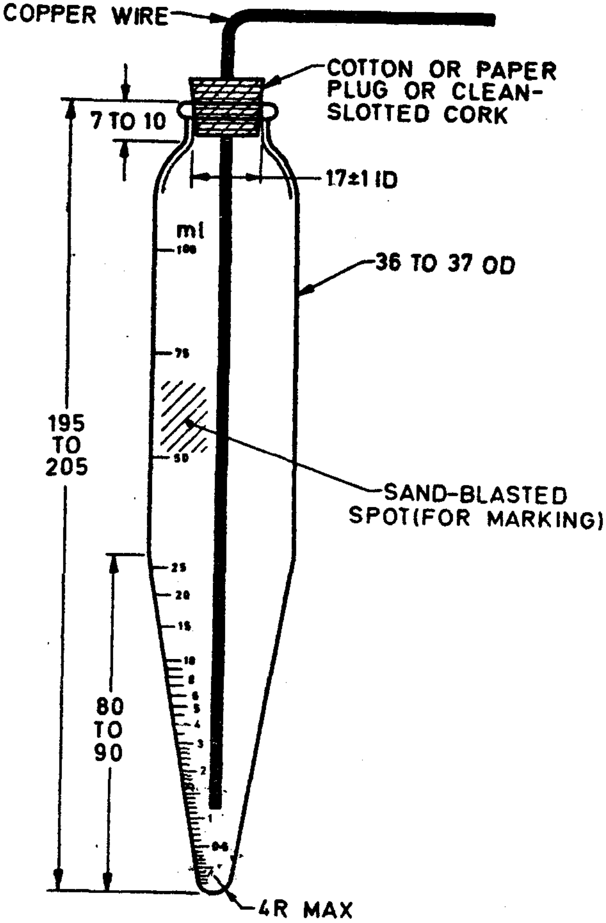

All dimensions in millimetres.

FIG. 1 CENTRIFUGE TUBE

upward from the bottom of the tube. The values of the divisions for each range with limits of error for the concerned range are given in Table 1.

TABLE 1 LIMITS OF ERROR FOR THE CENTRIFUGE TUBE

RANGE (1) ml	SCALE DIVISION (2) ml	LIMIT OF ERROR FOR THE RANGE (3) ml	NUMBERED (4) ml
0 to 0·1	0·05	±0·02	—
0·1 " 0·3	0·05	±0·03	—
0·3 " 0·5	0·05	±0·05	0·5
0·5 " 1·0	0·1	±0·05	1
1 " 2	0·1	±0·1	2
2 " 3	0·2	±0·1	3
3 " 5	0·5	±0·2	4,5
5 " 10	1·0	±0·5	6,8,10
10 " 25	5·0	±1·0	15,20,25
25 " 100	25·0	±1·0	50,75,100

4.1.1 The centrifuge tube shall contain a copper wire as specified in **4.8**.

4.2 Sample Pre-cooling Equipment — having a cooling coil approximately 6 m long of about 6 mm OD soft copper tubing wound to a diameter of about 65 mm as shown in Fig. 2, assembled in a suitable cooling bath.

4.3 Syringe — 1-ml ordinary medical syringe, graduated in 0·1 ml and equipped with a needle 200 mm long or, alternatively, a 1-ml pipette graduated in 0·1 ml.

4.4 Thermometer — conforming to Schedule 2 of IS : 2480-1964*.

4.5 Water-Bath — A 250-ml beaker is suitable as a water container.

4.6 Filter Paper — medium-grade, rapid, white, 12 cm in diameter.

4.7 Solvent Wash Bottle

4.8 Copper Wire — 2·00 mm in diameter and 300 mm long.

4.9 Solvent — Oil-free *n*-pentane. Cyclopentane or *n*-hexane may be used where the ambient temperature of altitude is too high for convenient handling of *n*-pentane.

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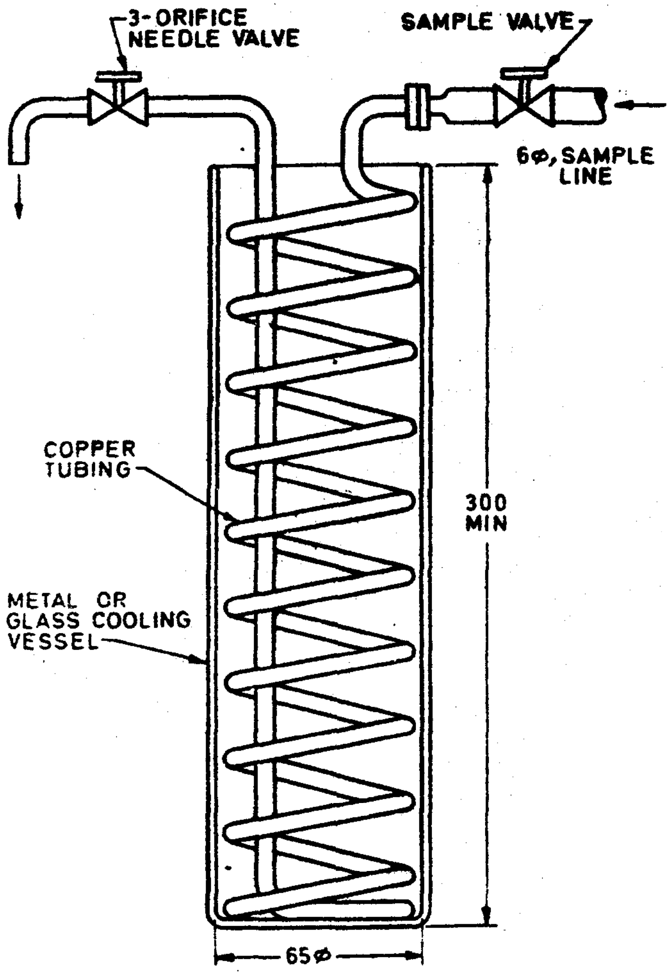
*Specification for general purpose glass thermometers.



All dimensions in millimetres.

FIG. 2 SAMPLE PRE-COOLING EQUIPMENT

4.10 Clamp — for holding the centrifuge during tube charging and weathering.

5. PREPARATION OF APPARATUS

5.1 Wash with the solvent all glassware to be used in the test. Add 10 ml of a new sample of solvent to the centrifuge tube. Mark the centre of the filter paper. Fill the syringe or pipette with a portion of the solvent drawn from the centrifuge tube and direct 0·1 ml of the solvent to the mark on the paper (*see* note under **5.2**). Allow the solvent to evaporate and note the persistence of an oil ring by holding the dry filter paper between the eye and a bright incandescent light or strong daylight through a window.

5.2 Make subsequent 0·1-ml additions until 1·5 ml of solvent has been added.

NOTE — The solvent is added in 0·1-ml increments to confine the solvent ring to a circle of about 30 mm in diameter. During the solvent addition filter paper should be held level by placing it on a 250-ml beaker.

5.3 If no oil ring appears after 1·5 ml of solvent has been added, the solvent and glassware are satisfactory. The appearance of an oil ring indicates either improperly cleaned glassware or contaminated solvent.

6. PROCEDURE

6.1 Residue — Attach the cooling coil to the sample source, cool the coil to a temperature below the boiling point of the sample, and flush the coil and sampling line. Rinse the centrifuge tube with the material to be sampled and then fill it to the 100-ml mark with a representative sample. Immediately insert the copper wire through a clean, slotted cork or a clean, loose-fitting plug of cotton or cleansing tissue in the mouth of the centrifuge tube. The wire helps to prevent superheating and resulting bumping (erratic or excessive boiling), and the cork (or plug) keeps out air or moisture while the sample is weathering. If more than 10 ml of the sample is lost because of bumping, obtain a new sample. Allow the sample to weather, using artificial heating, if the ambient temperature or type of sample require it.

CAUTION : *Safe means for disposal of products of weathering should be provided.*

If, when weathering has ceased and the tube has reached ambient temperature, a residue remains, place the tip of the tube in a water-bath at 38°C for 5 minutes.

NOTE — A satisfactory alternative to the use of thermometer and a 38°C bath is to warm the tip of the centrifuge tube with the hand.

6.1.1 Record the volume of any remaining residue to the nearest 0·05 ml.

6.2 Residue Number — Multiply the volume of residue retained in **6.1.1** by 200 and record the result as the observed *R* Number.

6.3 Oil Stain Observation — Add sufficient solvent to the centrifuge tube containing the residue described in **6.1.1** to restore the volume to 10 ml. Add the solvent from wash bottle (*see* Note 1) and carefully wash down the sides of the tube. Stir well with the syringe needle or pipette so that any residue at the bottom of the tube is dissolved uniformly in the solvent. Mark the centre of a clean white filter paper. Fill the syringe or pipette and direct 1·5 ml of solvent-residue solution at a rate such that the wetted circle is maintained at about 30 mm in diameter. If no oil ring persists after 2-minute waiting period when holding the dry filter paper between the eye and bright incandescent light, or strong daylight through a window, discontinue the test. If a ring is discernible, determine the volume of the solvent-residue solution at which the oil ring first persists for 2 minutes on a new filter paper by adding the solvent-residue solution in 0·1-ml increments (*see* Note 2).

NOTE 1 — Storage of oil-free solvent in the polyethylene wash bottle for several days contaminates the solvent. Any solvent transferred to the wash bottle for purposes of running the test, shall either be used in testing on same day or discarded.

NOTE 2 — It has been noted that at low ambient temperatures (below 4·4°C) materials in the gasoline boiling range have an oil ring that persists after 2 minutes. Oil ring determinations should be made in the protected area where the temperature is above 4·4°C. If it is necessary to determine the oil ring at temperatures of 4·4°C and below, allow 10 minutes for oil ring persistence.

6.3.1 Record the volume in millilitres of the solvent-residue solution required to yield a persistent oil ring as the oil stain observation.

6.4 O Number — Divide 10 by the oil stain observation obtained in **6.3.1** and record the result as the observed *O* Number. If no oil ring was discernible on adding 1·5 ml of solvent-residue solution in **6.3**, record the *O* Number as zero.

7. CALCULATION

7.1 Depending upon the requirements, report the result of test in terms of residue on evaporation and oil stain observation or, alternatively, in terms of residue number and oil number.

8. PRECISION

8.1 Results of duplicate tests shall not differ by more than the following amounts:

Oil Number	*Repeatability*	*Reproducibility*
0 to 20	4	6
Over 20 „ 40	6	8
Over 40 „ 100	8	12
Residue Number		
0 to 20	5	10
Over 20 „ 40	10	10
„ 40 „ 60	20	30

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